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# EFFECT OF ALLOYING TYPE AND LEAN SINTERING ATMOSPHERE ON THE PERFORMANCE OF PM COMPONENTS

M. Vattur Sundaram, R. Shvab, S. Millot, E. Hryha, L. Nyborg

## Abstract

*In order to be cost effective and to meet increasing performance demands, powder metallurgy steel components require continuous improvement in terms of materials and process development. This study demonstrates the feasibility of manufacturing structural components using two different alloys systems, i.e. lean Cr-prealloyed and diffusion bonded water atomised powders with different processing conditions. The components were sintered at two different temperatures, i.e. 1120 and 1250 °C for 30 minutes in three different atmospheres: vacuum, N<sub>2</sub>-10%H<sub>2</sub> atmosphere as well as lean N<sub>2</sub>-5%H<sub>2</sub>-0.5%CO-(0.1-0.4)%CH<sub>4</sub> sintering atmosphere. Components after sintering were further processed by either low pressure carburizing, sinterhardening or case hardening. All trials were performed in the industrial furnaces to simulate the actual production of the components. Microstructure, fractography, apparent and micro hardness analyses were performed close to the surface and in the middle of the sample to characterize the degree of sintering (temperature and atmosphere) and the effect of heat treatment. In all cases, components possess mostly martensitic microstructure with a few bainitic regions. The fracture surface shows well developed sinter necks. Inter- and trans-granular ductile and cleavage fracture modes are dominant and their fraction is determined by the alloy and processing route.*

**Keywords:** *lean alloyed PM steels, lean sintering atmospheres, low pressure carburizing, sinterhardening, case hardening*

## INTRODUCTION

In order to stay competitive against conventional structural component manufacturing processes in terms of both, cost and performance, powder metallurgy (PM) steel components require improved material and processes. Alloying elements such as Ni, Mo, and Cu, mostly used in conventional PM steels nowadays, are rather expensive. The introduction of Cr by prealloying is a cost-effective alternative to replace the current alloying systems due to its low cost and excellent recyclability in comparison to e.g. Cu-containing PM steels. However, industrial utilization of an alloy with Cr brings up challenges due to its affinity towards oxygen and risk of stable oxides formation. These stable oxides inhibit the early formation of inter-particle necks during sintering and thereby affect the strength of the material [1,2].

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Hence, sintering of Cr-prealloyed PM steels require proper sintering conditions, preferably high-temperature sintering in a reducing and controlled sintering atmosphere in order to assure efficient reduction of the oxides. Hydrogen in the sintering atmosphere is crucial to reduce the surface iron oxide at lower temperatures between 350 to 550 °C [3-6]. Carbon, typically added as graphite, plays a major role in the further reduction of stable oxides at temperatures around 750 °C [4, 7]. This reduction combination provides a suitable sintering condition for oxide reduction and growth of inter-particle necks.

Chromium alloying introduces ferrite hardening affecting the compressibility but when introduced in the lower amount rather good compressibility can be reached [8]. Recent studies performed on the same lean alloy as used in this study (Fe-1.8 wt.% Cr) show that oxide reduction and transformation are taking place at the same temperature range as mentioned earlier [9, 10], confirming the need for hydrogen containing atmosphere for early reduction of the surface iron oxide layer [9]. Karmachedu *et.al* has shown that the sintering of Cr-prealloyed PM steels using lean sintering atmospheres containing hydrogen, carbon monoxide and hydrocarbon for carbon control provides carburising effect [11]. For sintering of Cr-prealloyed PM steels, vacuum sintering seems to be another attractive option owing to the possibility to combine the efficient oxide reduction [6] and low-pressure carburising in one sintering process utilizing modern vacuum sintering furnaces [12].

In this work, the sintering of PM components is demonstrated through performing industrial trials at 1120 and 1250°C in various facilities. Sintering was performed using  $N_2$ -10H<sub>2</sub>,  $N_2$ -5H<sub>2</sub>-0.5CO-(0.1-0.4) CH<sub>4</sub> and vacuum combined with suitable heat treatments to meet the component specific requirements. The advancement in the sintering process and technologies are well-known for combining sintering and hardening into sinter hardening and introducing low-pressure carburising after vacuum sintering. By taking this into consideration and based on the proposed atmospheres for sintering of Cr-prealloyed PM steels, the effectiveness of different sintering atmospheres and heat treatments are evaluated through the basic characterization of the components.

## MATERIALS AND METHODS

Water atomised powders, prealloyed with 1.8 wt.% Cr (Astaloy CrA) and experimental low Cr alloyed powder were used for the major portion of the study. They were admixed with 2 wt.% Cu, 0.45 and 0.65 wt.% graphite, respectively, and 0.75 wt.% Lube E lubricant. For the one trial, diffusion bonded powder (Distaloy DH) which contains 1.5 wt.% Mo prealloyed and diffusion bonded with 2 wt.% Cu was used as well. All the powder mixes were from Höganäs AB, Sweden, and were used for manufacturing the test components. Components were compacted at FJ Sintermetal AB, Sweden. Different sintering atmospheres such as  $N_2$ -10H<sub>2</sub> (at FJ Sintermetal AB),  $N_2$ -5H<sub>2</sub>-0.5CO-(0.1-0.4)CH<sub>4</sub> (at AGA-Linde, Munich) were utilized and also sintering in a vacuum (at Höganäs AB) was performed at 1120°C for 30 min as given in Table 1 and Table 2. Metallography, hardness and fractography studies were performed on sintered samples.

Tab.1. Components sintered under different conditions and the designations used

Material	Sintering temperature, °C – time, min	Sintering atmospheres		
		N <sub>2</sub> -10%H <sub>2</sub>	N <sub>2</sub> -5%H <sub>2</sub> -0.5%CO-(0.1-0.4)%CH <sub>4</sub>	Vacuum
Astaloys CrA + 2 wt.% Cu + 0.45 wt.% C + 0.75 wt.% Lube E	1120-30	✓	✓	✓
Low Cr alloyed + 2 wt.% Cu + 0.65 wt.% C + 0.75 wt.% Lube E	1120-30	✓	✓	✓
Distaloy DH + 0.6 wt.% C + 0.6 wt.% Lube E	1120-30	–	✓	–

Tab.2. Heat treatment following the different sintering atmospheres

Sintering atmosphere	Heat treatment
N <sub>2</sub> -10%H <sub>2</sub>	Carbonitriding
N <sub>2</sub> -5%H <sub>2</sub> -0.5%CO-(0.1-0.4)%CH <sub>4</sub>	Sinterhardening
Vacuum	Low pressure carburising

## RESULTS AND DISCUSSION

### Metallographic investigation

Figure 1 shows the microstructure of Astaloy CrA+2Cu+0.45C material sintered at different atmospheres and corresponding heat treatments.

Pore distribution and size was found to be quite similar for all the samples based on Astaloy CrA+2Cu+0.45C material. Microstructure in all the cases is martensitic. Some small areas of retained austenite were observed for vacuum sintered + low pressure carburised samples.

Low Cr alloyed material sintered at the same sintering atmospheres and heat treatments, showed mostly martensitic microstructure with the presence of bainite, see Fig.2.

Distaloy DH material was sintered in N<sub>2</sub>-5H<sub>2</sub>-0.5CO-(0.1-0.4)CH<sub>4</sub> atmosphere with applied sinterhardening only. The respective microstructure is shown in Figure 3. It is fully martensitic at both surface (Fig. 3a) and the centre (Fig. 3b) of the sample.

Apparent hardness and microhardness for the investigated materials are summarised in Table 1. Relatively high values of hardness were achieved by application of all the sintering processes to the selected in the investigation materials. In most cases, materials were through hardened. Vacuum sintering + low pressure carburisation of Astaloy CrA+2Cu+0.45C material showed a significant difference in microhardness between surface and centre indicating case hardening.

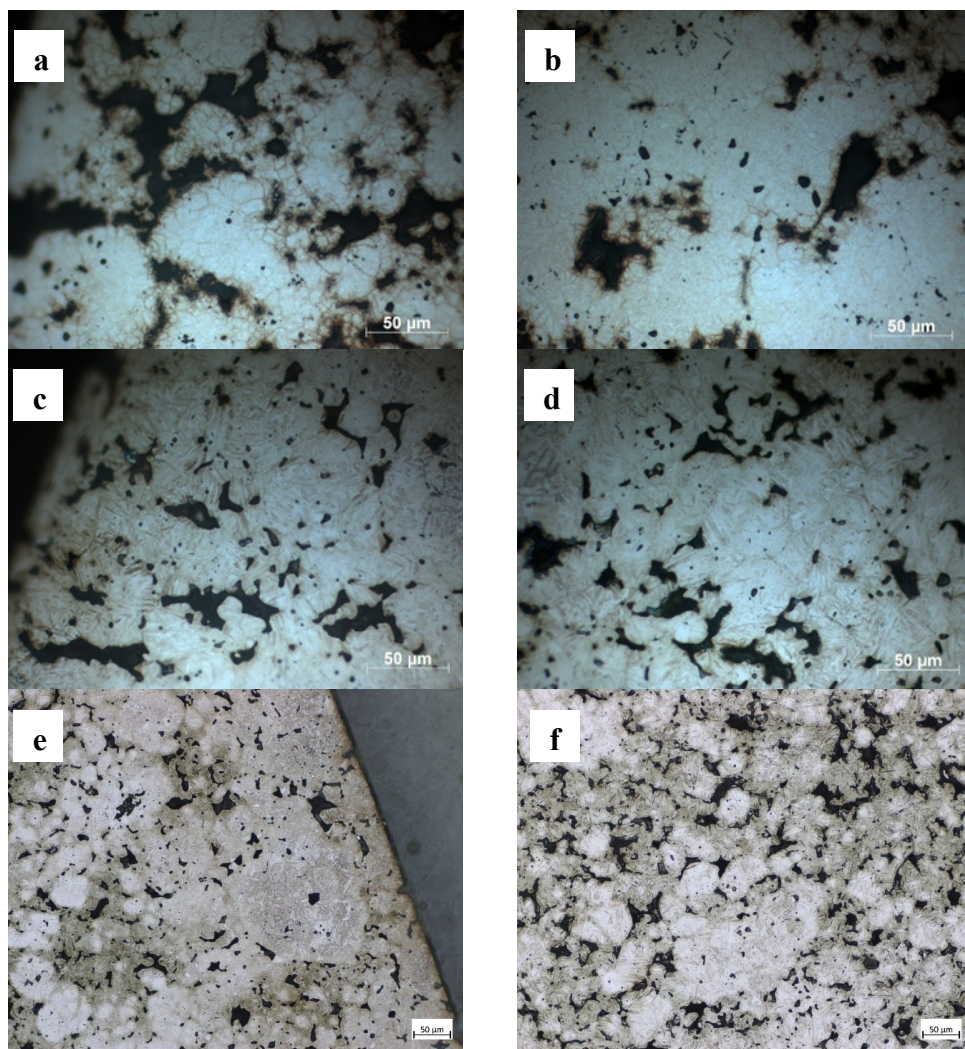


Fig.1. Optical micrographs of Astalloy CrA+2Cu+0.45C material sintered at N<sub>2</sub>-10H<sub>2</sub> + carbonitriding (a & b), N<sub>2</sub>-5H<sub>2</sub>-0.5CO-(0.1-0.4) CH<sub>4</sub> + sinterhardening (c & d), sintered in vacuum + low pressure carburising (e & f).



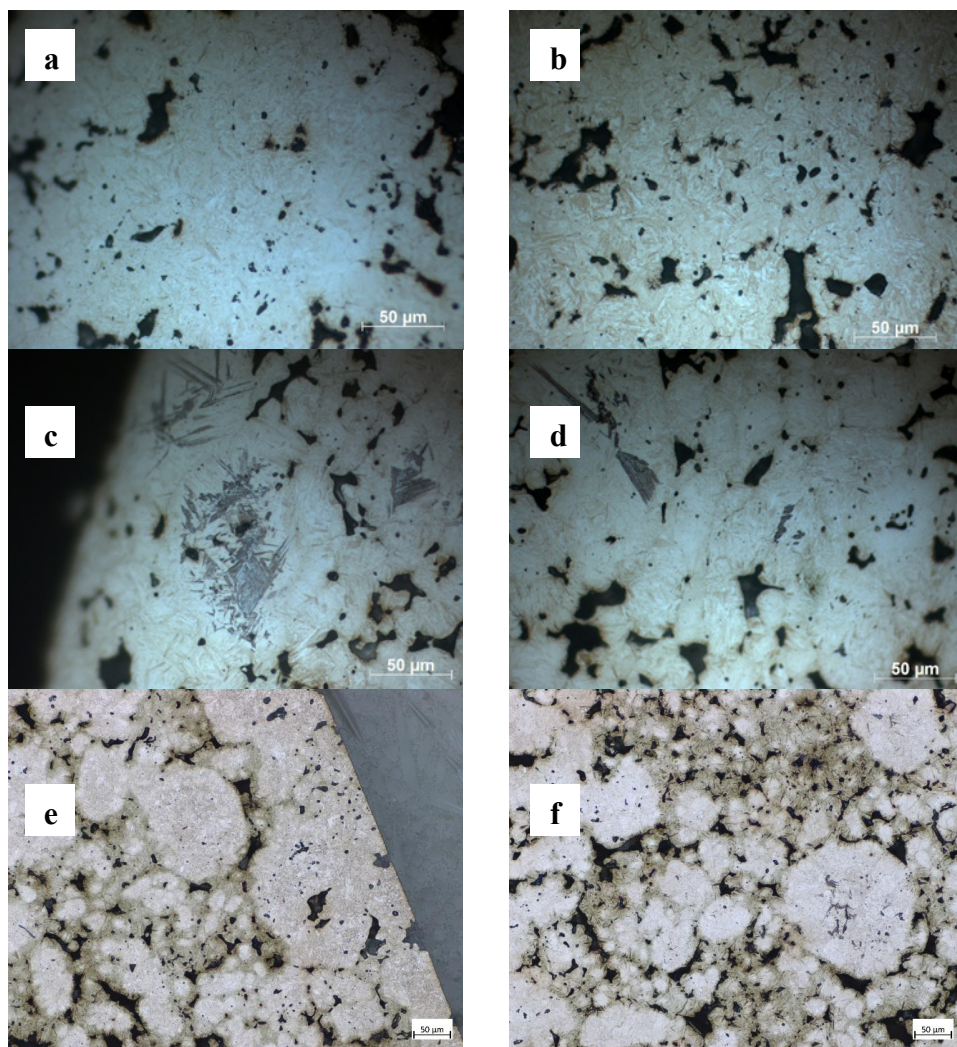


Fig.2. Optical micrographs of low Cr alloyed material sintered at  $N_2-10H_2$  + carbonitrided (a & b),  $N_2-5H_2-0.5CO-(0.1-0.4) CH_4$  + sinterhardened (c & d), sintered in vacuum + low pressure carburised (e & f).

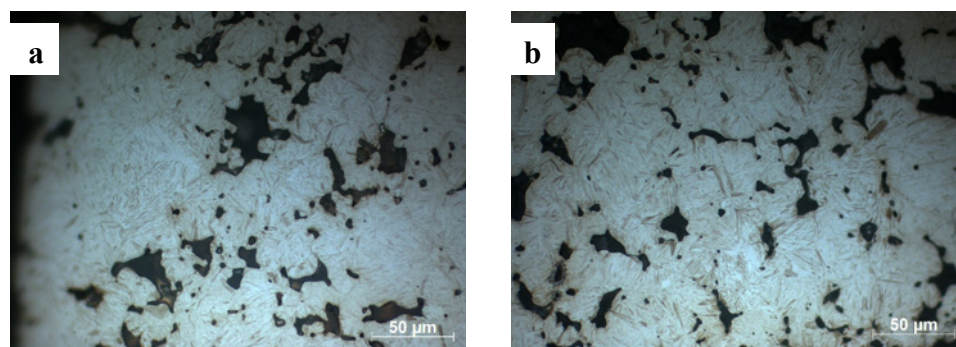


Fig.3. Optical micrographs of Distaloy DH material sintered at  $N_2$ -5H<sub>2</sub>-0.5CO-(0.1-0.4)CH<sub>4</sub> and sinter-hardened.

Tab. 2. Apparent hardness and microhardness of investigating materials

Material	Process	Hardness HV5		Microhardness HV0.3	
		Surface	Centre	Surface	Centre
Astaloy CrA+2Cu+ 0.45C	N <sub>2</sub> -10H <sub>2</sub> + carbonitriding	603±14	468±8	735±42	846±21
	N <sub>2</sub> -5H <sub>2</sub> -0.5CO-(0.1-0.4)CH <sub>4</sub> + sinterhardening	474±12	334±0	622±16	576±16
	vacuum + low pressure carburising	-	-	820±10	550±10
Low Cr alloyed+2C u+0.75C	N <sub>2</sub> -10H <sub>2</sub> + carbonitriding	602±30	413±22	740±31	571±17
	N <sub>2</sub> -5H <sub>2</sub> -0.5CO-(0.1-0.4)CH <sub>4</sub> + sinterhardening	485±17	501±10	584±5	678±12
	vacuum + low pressure carburising	-	-	830±10	700±10
Distaloy DH+0.6C	N <sub>2</sub> -5H <sub>2</sub> -0.5CO-(0.1-0.4)CH <sub>4</sub> + sinterhardening	446±7	353±24	613±11	658±4

### Fracture surface analysis

Fracture surface analysis was carried out to analyse the failure mechanisms of investigated materials and detect the possible ways to improve material properties. The focus was placed on components sintered with N<sub>2</sub>-10H<sub>2</sub> + carbonitriding and N<sub>2</sub>-5H<sub>2</sub>-0.5CO-(0.1-0.4)CH<sub>4</sub> + sinterhardening processes.

Figure 4 shows the appearance of Astaloy CrA+2Cu+0.45C material sintered at N<sub>2</sub>-10H<sub>2</sub> + carbonitriding and N<sub>2</sub>-5H<sub>2</sub>-0.5CO-(0.1-0.4)CH<sub>4</sub> + sinterhardening processes. In the both cases, intergranular decohesion is the dominant mechanism of failure throughout the components, see Figure 4. EDX analyses made on the fracture surface of the same materials indicate the inhomogeneous distribution of Cu throughout the material, see an example in Figure 5. Oxide free pore surfaces were observed in the case of N<sub>2</sub>-5H<sub>2</sub>-0.5CO-(0.1-0.4)CH<sub>4</sub> + sinterhardening process. Oxides, observed inside the pores of Astaloy CrA, sintered in N<sub>2</sub>-10H<sub>2</sub> + carbonitrided can be the consequence of post treatment.

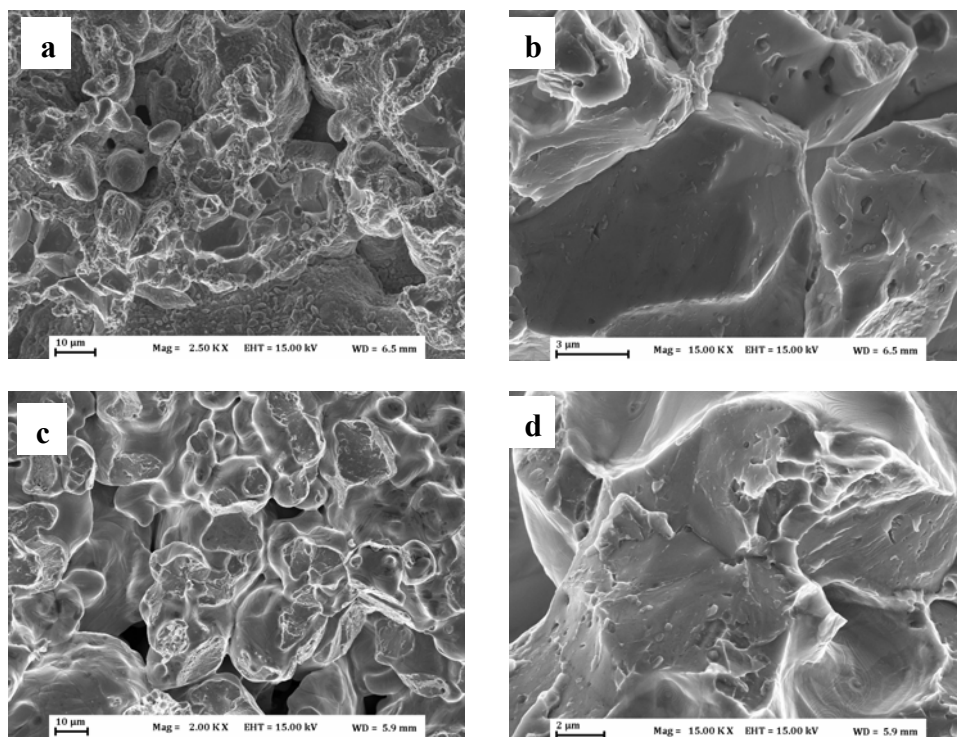
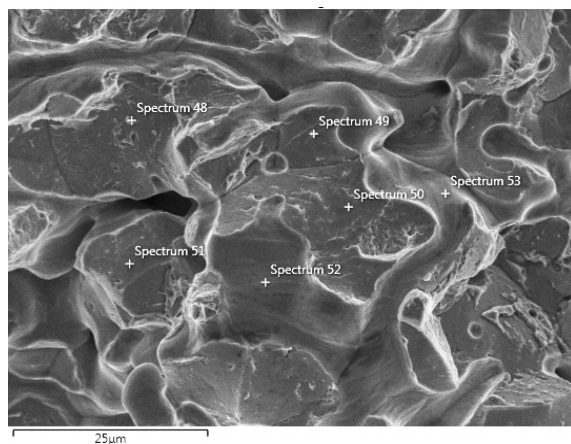


Fig.4. Fracture surface of Astaloy CrA+2Cu+0.45C material sintered at  $N_2$ -10H<sub>2</sub> + carbonitriding (a & b) and  $N_2$ -5H<sub>2</sub>-0.5CO-(0.1-0.4)CH<sub>4</sub> + sinterhardened (c & d).



Spectrum Cu	wt. %
48	4.8
49	4.9
50	6.5
51	7.4
52	7.3
53	7.9

Fig.5. EDX analysis of Astaloy CrA material sintered at  $N_2$ -5H<sub>2</sub>-0.5CO-(0.1-0.4)CH<sub>4</sub> + sinterhardening process.

In the case of low Cr alloyed material, samples sintered at both  $N_2$ -10H<sub>2</sub> + carbonitriding and  $N_2$ -5H<sub>2</sub>-0.5CO-(0.1-0.4)CH<sub>4</sub> + sinterhardening processes are characterized by ductile and cleavage fracture, see Figure 6.



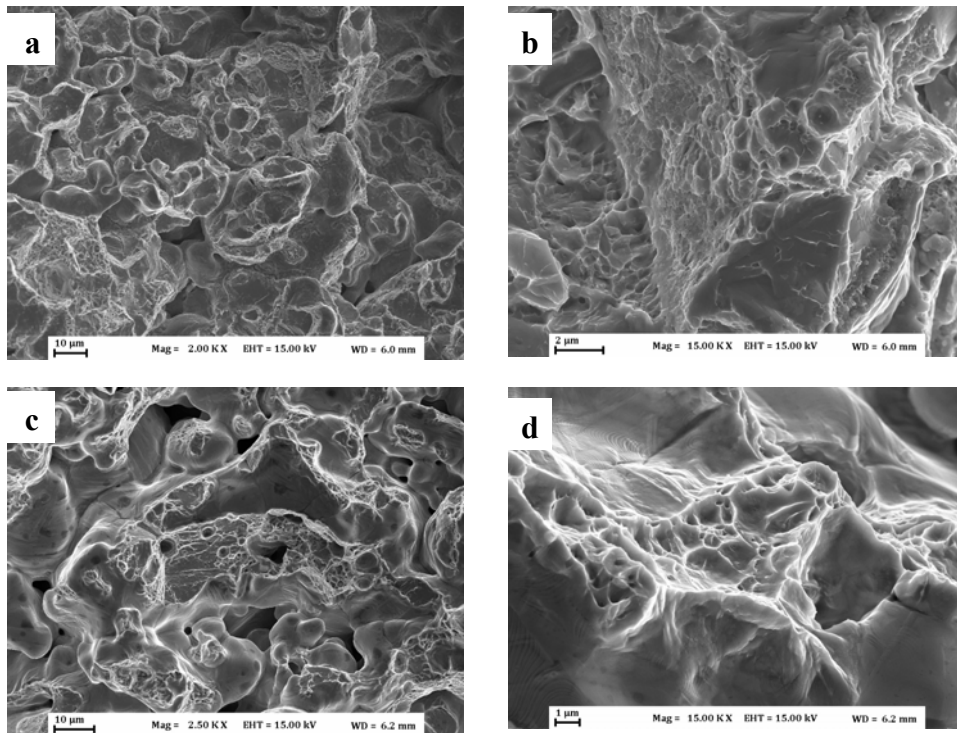
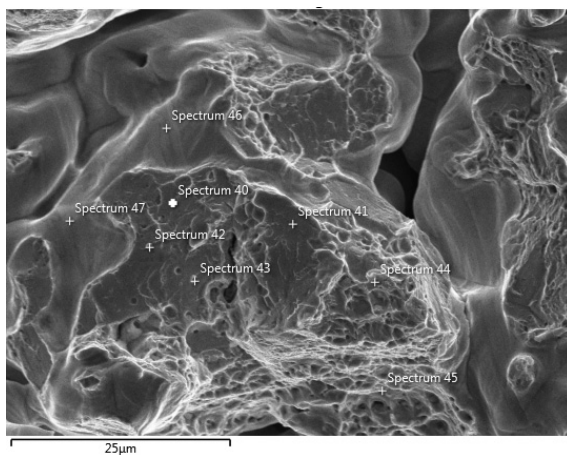


Fig.6. Fracture surface of low Cr alloyed material sintered at  $N_2-10H_2$  + carbonitriding (a & b) and  $N_2-5H_2-0.5CO-(0.1-0.4)CH_4$  + sinterhardening (c & d) processes.



Spectrum Cu	wt.%
40	6.9
41	6.4
42	5.0
43	0.3
44	0.3
45	0.0
46	8.6
47	0.2

Fig.7. EDX analysis of low Cr alloyed material sintered at  $N_2-5H_2-0.5CO-(0.1-0.4)CH_4$  + sinterhardening process.

Similarly to Astaloy CrA material, distribution of Cu in the low Cr alloyed material is also not even, see Fig.7. This can lead to material's weakening and might be avoided by adjusting the sintering process (increasing temperature or time of sintering) which will provide better distribution of Cu in the material. Pore surface quality of low Cr

alloyed material is the same as for Astaloy CrA and is the consequence of the applied sintering process.

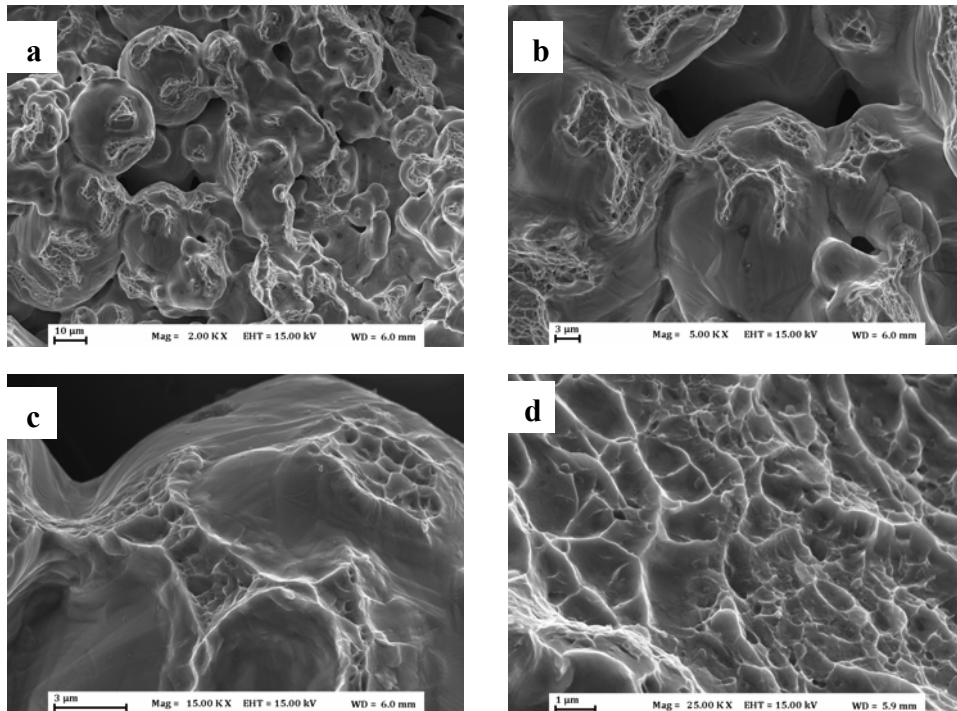


Fig.8. Fracture surface of Distaloy DH material sintered at  $N_2-5H_2-0.5CO-(0.1-0.4)CH_4$  + sinterhardening process.

The ductile interparticle fracture was found to be the main failure mechanism of the Distaloy DH material sintered at  $N_2-5H_2-0.5CO-(0.1-0.4)CH_4$  + sinterhardening process, see Fig.8. Oxide free pore surfaces and inter-particle connections indicate the high quality of sintering process.

## CONCLUSIONS

Based on all the three processes, the demonstrated Cr-prealloyed materials show the ability to replace the diffusion bonded material for the intended application. Among the proposed alternatives, sintering in  $N_2-10\%H_2$  followed by carbonitriding showed the highest hardness, but also indicated higher oxidation and embrittlement. Components sintered in the lean atmosphere followed by sinterhardening showed lower surface and core hardness, but with much better inter-particle connections and overall component strength. Sintered in vacuum followed by low pressure carburising of the components allowed to reach high surface hardness level as in case of carbonitrided components with good overall component strength and degree of sintering. Hence, utilization of the lean Cr-alloy PM-steels and proper sintering and post-sintering treatments allows to meet the performance standards and improve cost competitiveness of the components. This enables a flexible processing route for the realising the final requirements of a PM component. All these combinations give a tremendous advantage when sintering large volume of parts in an industrial scale.

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## REFERENCES

- [1] Karlsson, H., Nyborg, L., Berg, S.: Powder Metall., vol.48, 2005, p. 51
- [2] Chasoglou, D.: Ph.D. Thesis. Chalmers University of Technology, 2012
- [3] Hryha, E. et.al: Appl. Surf. Sci., vol. 256, 2010, p. 3946
- [4] Hryha, E., Dudrova, E., Nyborg, L.: J. Mater. Process. Technol., vol. 212, 2012, p. 977
- [5] Chasoglou, D., Hryha, E., Nyborg, L. In: World PM 2010. Vol. 2, 2010, p. 3
- [6] Chasoglou, D., Hryha, E., Nyborg, L.: Mater. Chem. Phys., vol. 138, 2013, p. 405
- [7] Danninger, H. et.al: Powder Metall. Prog., vol. 2, 2002, p. 125
- [8] Hryha, E., Dudrova, E., Bengtsson, S.: Powder Metall., vol. 51, 2008, p. 340
- [9] Vattur Sundaram, M. et.al: Powder Metall. Prog., vol. 14, 2014, p. 85
- [10] Hryha, E., Nyborg, L.: Metall. Mater. Trans. A, vol. 45, 2014, p. 1736
- [11] Karamchedu, S.: Ph.D. Thesis. Chalmers University of Technology, 2015
- [12] Vattur Sundaram, M. et.al In: Proc. World PM 2016-Steel Sintering, 2016